Specifications for a Combination High Vacuum and Controlled Atmosphere Furnace

Introduction

The Naval Research Laboratory, (NRL), requires a high temperature furnace (1700 degrees Celsius) which is capable of operating in three different furnace-chamber atmospheres: (1) high vacuum reaching 10^{-6} torr (1e-6 torr), (2) high purity gaseous hydrogen at 2 PSIG (pounds per square inch, gauge), and (3) high purity inert gaseous nitrogen at 2 PSIG. This furnace will be used for several extremely high purity assembly (less than 0.5 parts per million (ppm) water vapor) and processing steps needed in the construction of high power millimeter-wave and microwave vacuum electronic devices. These include the heat treating, brazing, and outgassing of metallic parts including stainless steel and copper, and the firing and active-metal-based metalization of advanced technical ceramics such as aluminum oxide, silicon nitride, diamond, and aluminum nitride. The furnace system must meet or exceed the following specifications:

I. Mechanical and Thermal Specifications of the Furnace Chamber

1.1 Furnace Chamber Construction: The body of the chamber shall be cylindrical in shape and made from double wall, non-magnetic stainless steel, with flowing water cooling in the space between the steel layers. The inner wall shall have a minimum thickness of 3/16 inch, and the outer wall shall have a minimum thickness of 1/8 inch. The chamber shall be designed with baffles between the inner and outer walls so as to ensure uniform cooling of the chamber. The top and bottom chamber covers and the access lid shall be made from either water cooled high-strength aluminum alloy, or water-cooled nickel-plated copper, or water-cooled non-magnetic stainless steel. Permanent seals between furnace chamber parts, as well as any seals between the furnace chamber and areas containing water, must all be TIG (Tungsten-Inert-Gas) welded. In demountable joints between various chamber parts not associated with water, the O-ring seals must be grease-free Viton or equivalent. The furnace chamber must be designed per the NFPA (National Fire Protection Association) code.

1.2 Required Operating Temperature: The furnace shall operate at a minimum of 1700 degrees C (degrees Celsius)

1.3 Required Atmospheres: The furnace shall reach a minimum of 10^{-6} torr (1e-6 torr) level vacuum; dry hydrogen at 2PSIG; dry nitrogen at 2PSIG.

1.4 Hot Zone Heating Method and Materials: The furnace shall be heated by means of an electrically energized heating element located inside the furnace chamber. The heating element shall consist of six (6) tungsten mesh panels, arranged to form a cylinder surrounding the work zone. Tungsten weave panels are also acceptable. The heating element shall be suspended by tungsten rods or bars, which also function as the electrical connections. The hot zone must be insulated from all of the furnace chamber walls by means of layered refractory metal heat shields, made from either molybdenum or tungsten (or a combination).

IMPORTANT NOTE: The use of any of the following materials in any of the furnace's internal construction will be considered technically unacceptable: graphite, moly-disilicide ($MoSi_2$), porous-ceramics, firebrick, ceramic-fiber-materials, silicon-carbide, or metals with high vapor pressure constituents (zinc, cadmium, lead, antimony, arsenic, selenium, tellurium, mercury).

1.5 Hot Zone Heating Element Size: Cylindrical; 8.0 inches diameter by 18.0 inches high

1.6 Working Area: This refers to the usable internal working volume of the furnace, within the hot zone heating element. This allows for clearances between the heating element and the work piece under processing. The usable working volume inside the furnace shall be no less than 7.0 inches diameter by 14.0 inches high.

1.7 Loading Method: The furnace shall be designed for top loading, with access provided by a top-mounted, hinged, O-ring sealed hatch. The hatch must be securable with hand tightenable bolts or clamps. The diameter of the hatch opening shall be at least 7.5 inches to allow easy access to the working area, and there shall be no immovable restrictions or blockage between the hatch opening and the working area that would reduce the usable working area diameter below 7.0 inches.

1.8 Heat Shield Geometry: The refractory metal heat shield pack located between the heating element and the water-cooled furnace chamber vessel shall consist of a minimum of five (5) concentric cylinders of refractory metal sheets (either tungsten or molybdenum, or a combination of the two types, with each sheet having 0.005 inch minimum thickness), separated from each other by coiled refractory metal wire, and bound into an assembly sufficiently robust to prevent shorting to the heating element. Likewise, the bottom heat shields, located on the floor of the furnace, shall consist of a similarly constructed and bound stack of a minimum of five (5) refractory metal sheets, each sheet having a 0.005 inch minimum thickness. The top shields shall consist of a removable stack of similarly bound and spaced refractory metal sheets, to insulate the access door from the hot zone (at least five (5) sheets, each a minimum of 0.005 inches thick). Finally, the regions where the electrical feedthroughs for the heating elements enter the hot zone as well as the location of the pumpout port shall be insulated with stacks or baffles of refractory metal sheets of similar construction.

1.9 Work Support: The furnace must have a tungsten work support plate of 3/16 inch minimum thickness located inside the heating element area. This plate must be hexagonal or circular in shape and shall be supported from below by six (6) tungsten rods. The plate and rods shall be removable through the access port for service or for changing the support rods. The furnace shall be shipped with two plates and two different sets of tungsten support rods. One set of six rods will have a length that position the plate 2 inches up from the bottom of the heating element. The second set of six rods will be long enough to position the plate in the middle of the heating element (9 inches from the element bottom). Thus, a total of twelve (12) rods and two (2) interchangeable work support plates are required.

1.10 Additional Requirements: The overall design of the heating elements and heat shields shall result in the center 2/3 of the hot zone exhibiting a temperature uniform to ±10 degrees C, when measured along the vertical central axis of the furnace. The furnace must have a viewing port. This window must have a <u>viewable</u> diameter into the hot zone of no less than 3/8 inch, and no greater than 1/2 inch. The viewing port shall be placed halfway up the chamber, in the front. The electrical power feedthroughs must be water cooled. The temperature of the outer jacket of the furnace shall not exceed the normal range of ambient temperatures (35 degrees C max) under any circumstances.

II. Vacuum System / Gas Handling System

2.1 Required Vacuum Levels: The furnace shall be equipped with a vacuum system capable of producing vacuums in the 10^{-6} torr (1e-6 torr) range. The attainable vacuum shall be better than $3x10^{-6}$ torr (3e-6 torr) at 1700 degrees C and $1x10^{-6}$ torr (1e-6 torr) at room temperature.

2.2 Vacuum System Description: The high vacuum system shall consist of a rough pump in conjunction with a silicone-oil diffusion pump. A high-vacuum gate valve shall be located between the diffusion pump/cold trap assembly and the furnace chamber. A liquid nitrogen cold trap must be installed between the high vacuum valve and the diffusion pump to prevent backstreaming of oil into the furnace. <u>Only a liquid nitrogen trap is acceptable</u>. The diffusion pump inlet and inlet vacuum plumbing must have a diameter of at least 6 inches, and the diffusion pump must have a peak pumping rate of at least 2,300 liters per second for air and a peak throughput of at least 3.5 torr-liters/second. The liquid nitrogen cold trap must have a nominal conductance of at least 2,000 liters/sec for air, and a liquid nitrogen reservoir volume of a minimum of 10 liters. The retained pumping speed for the diffusion pump with the cold trap installed must be at least 1,000 liters per second for air. The roughing pump must be a double-stage rotary-vane mechanical pump with a free air displacement of at least 25 cfm and an ultimate partial pressure below $1x10^{-3}$ torr (1e-3 torr). The pumping system must have a copper gauze roughing/foreline trap located between the roughing pump and the diffusion pump to prevent contamination. The pumping system must be equipped with an ionization gauge and gauge controller to permit accurate measurements of the high vacuum in the furnace chamber. In addition, thermocouple-style gauges must be placed on both the foreline and furnace chamber to monitor the status of the system during pumpdown.

2.3 Gas Handling System: The furnace must be able to withstand a 2 PSIG positive pressure for operation in the dry hydrogen and nitrogen atmospheres. The chamber must be equipped with a mechanical, compound vacuum/pressure gauge for pressure measurement. Gas admission must be controllable by a series of electric valves, in conjunction with flowmeters and needle valves for gas control. The flow meters must be able to accurately measure gas flows from 0 to 25 liters per minute. The furnace access lid must have clamps or bolts to keep it securely closed when under positive pressure. In addition, a relief valve must be included to prevent damage due to overpressurization. The furnace shall be supplied with a continuously operating hydrogen igniter and burnoff system to safely dispose of waste hydrogen gas or any other flammable exhaust that may exit the furnace chamber. The furnace must include a large diameter, spring loaded relief plate (a so called "safety blow-off port"), to safely vent any suddenly appearing pressures caused by an accidental hydrogen ignition within the furnace and therefore prevent catastrophic damage to the furnace and injury to personnel. The gas handling system and safety blow-off port must be designed per the NFPA code.

2.4 Vacuum and Gas Control and Safety Interlock System: All vacuum and gas valves must be electrically or electro-pneumatically controllable from switches on a control panel. The only exceptions to this are the flow adjustment controls on the gas flowmeters, which will be manually adjustable. The status of all electrically or electro-pneumatically controlled valves must be indicated with illuminated lamps on the control panel adjacent to the switches. Low pressure alarms must be included on the inlet gas lines. The vacuum valve sequencing during pumpdown must be automatically controlled and interlocked to prevent damage to the pumping system. The diffusion pump heater must have an interlock to prevent damage due to excessive temperatures or cooling water failure. All vacuum valves must be designed to close if the power fails. Furthermore, during a power failure, dry nitrogen must be automatically admitted into the furnace chamber at a <u>moderate</u> rate (max. 10 liters/minute) to bring the chamber to a safe status, including cases when hydrogen was present prior to power failure. The system must not allow hydrogen to be admitted into the furnace unless either (a) the vacuum system was operated first (to evacuate the chamber), or (b) the chamber was flushed with nitrogen for 15 minutes. Furthermore, during hydrogen operation a thermocouple must sense the presence of a hydrogen flame in the burnoff system, and shut off the hydrogen and purge with nitrogen at a moderate rate if the flame should accidentally extinguish. A mechanism to bypass this function during the initial filling of the chamber with hydrogen must be provided. A similar nitrogen purging safety sequence must occur if the low hydrogen inlet pressure alarm is triggered during a hydrogen processing run. Finally, the gas system must be designed to allow the operator to switch from hydrogen to nitrogen during furnace operation, to gradually expel the flammable gas. This deliberate operator-controlled transition to nitrogen must occur through the ordinary nitrogen flowmeter and valving, and is distinct from the automatic nitrogen purging which occurs in the event of a power failure, or an accidental extinguishing of the hydrogen burnoff flame, or a low hydrogen pressure alarm. The gas control and safety interlock system must be designed per NFPA codes.

III. Specifications on Main Furnace Power and Controls

3.1 Power Supply: The power supply system must be designed to operate from three-phase, 480 volt, 60 Hz AC power, at no more than 80 kVA. The power supply must be silicon-controlled rectifier (SCR) controlled, and must be capable of creating a maximum heating rate of 80 degrees C/minute. The power supply must be fused to prevent catastrophic damage due to an accidental short circuit. The cables connecting the power supply to the heater must be water cooled. The main control panel must have a voltmeter and ammeter calibrated to measure the voltage and current present in each phase of the heater. The power supply must be designed per NFPA codes.

3.2 Temperature Control: Automatic temperature control of the furnace using a microprocessor based proportional-integral-differential (PID) programmer/controller is required. The sensor must be located within the hot zone, and it must consist of a sheathed thermocouple appropriate for the temperature range (to 1700 degrees C) and all of the atmospheres discussed above. The thermocouple must be located halfway up the chamber and sealed to the chamber with a suitable compression fitting. This fitting must allow the end of the thermocouple to be placed at any appropriate radial position (the sensor movement will occur while a part is being set up in the furnace). The thermocouple must be capable of being completely withdrawn from the working area for inserting large objects or for service purposes. The controller must maintain the programmed temperature vs. time profile by a closed-loop feedback control to the power supply SCRs. The controller must be capable of following a program with at least 8 ramp and 8 soak (dwell) segments, with an absolute accuracy of at better than plus/minus 10 degrees C from 400 to 1700 degrees C, and plus/minus 15 degrees C at temperatures below 400 degrees C. With proper PID programming, the controller must be capable of maintaining a plus/minus 1 degree C relative temperature stability during an isothermal hold at any operating point from 200 to 1700 degrees C.

3.3 Power supply and Heater Interlocks: The flow of water in the cooling system of the furnace must be monitored, and the power to the hot zone must be automatically disconnected within five (5) seconds should the flow become insufficient for proper cooling. A similar interlock must be present for any water cooling that is needed by the main power supply. Should the power fail, the heater must not automatically re-energize upon resumption of power. There must also be an interlock to prevent excessive furnace temperatures. This device must automatically interrupt the heater power to prevent damage to the furnace or the work when an adjustable upper limit is reached. This interlock, which must be based around a separate thermocouple and limit controller, must be capable of overriding the main controller described in the previous section.

3.4 Miscellaneous Control Specifications: The furnace system must have a main breaker to permit quick, reliable power disconnection and for protection from serious malfunctions.

IV. Compatibility with Utilities

4.1 The proposed furnace system must be compatible with the following existing utilities: Power: 480V, 80 kVA, 3 phase AC power at 60 Hz; Water: filtered, 70 °F tap water at 70 psi at flows to 20 GPM; Air: 70 PSIG, filtered and lubricated. NRL also has regulated ultra high purity hydrogen (< 0.5 ppm water) and ultra high purity nitrogen, each at 20 PSIG.

V. Warranty

5.1 Offerors shall offer the Government at least the same warranty terms, including offers of extended warranties, offered to the general public in customary commercial practice.

VI. Documentation

6.1 NRL requires two (2) copies of an operators manual for the furnace, two (2) copies of the furnace chamber mechanical blueprints, two (2) copies of the gas system flow diagrams, and two (2) copies of the furnace electrical schematics. NRL also requires one (1) copy each of the manufacturers documentation for (a) the roughing vacuum pump, (b) the diffusion pump, (c) the vacuum ionization/thermocouple gauge controller, (d) the PID temperature controller, and (e) the over-temperature sensor/interlock. These requirements are separately identified in Exhibit A –Contract Data Requirements List (DD Form 1423).